

PROJECT NUMBER: 1752  
PROJECT TITLE: Optical Spectroscopy of Tobacco and Smoke  
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PERIOD COVERED: November, 1988

## I. TANDEM MASS SPECTROMETRY

- A. Objective: To establish a mass spectrometry center with the state of the art capability in tandem, high resolution, and soft ionization mass spectrometry.
- B. Results: In order to develop methodology for the characterization of complex lipids by utilizing soft ionization and tandem mass spectrometry, a trip was made to the Midwest Center for Mass Spectrometry. Lipid extracts from tobacco before and after ART processing, samples of residue from the ART process, and known lipid mixtures were prepared for FAB and NCI analysis. Some samples were analyzed directly. Others were fractionated into acid, basic and neutral fractions and in some cases simple derivitization was employed to enhance ionization. The samples were analyzed using the MS-50 triple analyzer mass spectrometer.
- C. Conclusions: Evaluation of data is in progress. Particularly significant results include information on fatty acid composition of the ART residues and differences in complex lipids in tobacco before and after the ART process. Work with known fatty alcohols demonstrated that nicotinyl chloride hydrochloride derivatives are useful for enhancing specific structural information for long chained alcohols.
- D. Plans: To evaluate the data of the ART residue samples and the known long chained alcohols. To prepare reports on data obtained and to explore some of the results with further experimentation.

## II. SMOKE ANALYSIS

- A. Objective: To compare the smoke chemistry of control and ART extracted filler.
- B. Results: Mainstream smoke from weight selected control (x6d8dej) and Art (x6d8dek) cigarettes were fractionated and compared to reveal any difference relating to the extraction process. Since the objective is to compare fillers, cigarette construction effects were eliminated by removing the CA filter and taping the ventilation holes prior to smoking. Mean area counts (from four replicates) from a set of target compounds in each fraction were compared. In the basic fraction, smoke from ART filler contained more volatile bases than that from control such as methylpyrazine; 2,6-dimethylpyrazine; 2,3,6-trimethylpyrazine; 3-methylpyrrolpyrazine; dimethylaminoacetonitrile, dimethylpyridines, ethylmethylpyridines, quinoline, and a minor alkaloid, nornicotine. The presence of these excessive bases was probably due to the high level of ammonia in the ART filler which

induced Maillard reaction with amino acids. The smoke from control showed higher amount of 3-vinylpyridine, 3-ethylpyridine, myosmine, nicotyrine, 2,2-dipyridine, cotinine, and some acyl alkaloids which reflected the difference in nicotine concentration between ART and control fillers. In the acidic fraction, volatile fatty acids such as 2-methylbutyric acid, valeric acid, beta-methylvaleric acids were reduced in the ART smoke. Some simple phenols such as cresols, guaiacol, 2-vinylphenol, and 4-vinylguaiacol were higher in the smoke of ART filler. Higher fatty acids showed comparable levels in both samples. In the neutral fraction, ART cigarette was consistently lower in the compounds identified such as furfural, limonene, 1-indanone, solanone, megastigmatrienones (4 isomers) and neophytadiene.

- C. **Conclusions:** The mainstream smoke of ART filler contained more volatile bases, some phenols, less volatile fatty acids, and less neutral compounds than that of control filler.

D. **References:**

Hsu, F, Coleman, S., and A. Oti, "Smoke Chemistry - ART," memo to B. Handy, October 31, 1988.

### III. THERMOGRAVIMETRIC ANALYSIS (TGA) OF CARBON DIOXIDE IN DOLOMITE

- A. **Objective:** The purpose of this experiment is to develop methodology for the determination of percent composition of carbon dioxide in the mineral Dolomite.
- B. **Results:** Six Dolomite samples were analyzed using a Perkin-Elmer TGA-7 analyzer. The samples were scanned from 25 C to 1025 C at a scan rate of 70 C/minute. The samples yielded from 31.0 percent to 31.5 percent weight loss from 421 C to 955 C which was assumed to be carbon dioxide since most carbonates decompose at this temperature range. The average weight of the carbon dioxide for the six samples was 31.3 percent.
- C. **Conclusions:** Based on the precision of the results, the relative short time necessary for analysis, and the ease of interpretation, TGA should be considered for the analysis of carbon dioxide in Dolomite and similar samples (i.e. samples containing carbonates and hydrates).
- D. **Plans:** The TGA method of analysis of carbon dioxide in Dolomite will be compared with previous methods. Future plans include structural elucidation and determination of percent composition of various carbonates and hydrates in Dolomite with data obtained from elemental analysis from Atomic Absorption and decomposition studies of Dolomite and pure carbonates and hydrates from TGA.

IV. THERMAL CHROMATOGRAPHY-MASS SPECTROMETRY

- A. Objective: To determine the effect of pet ether extraction on ART tobacco.
- B. Results: Comparisons are being made on feed filler, ART filler, and pet ether extracted ART filler using the TC/MS instrument.
- C. Conclusions: Data are under evaluation.